# 7. ENABLING TECHNOLOGIES

## A. Durability of Carbon-Fiber Composites

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### Objective

- Develop experimentally based, durability-driven design guidelines to ensure the long-term (15-year) integrity of representative carbon-fiber-based composite systems that can be used to produce large structural automotive components. Durability issues being considered include the potentially degrading effects of cyclic and sustained loadings, exposure to automotive fluids, temperature extremes, and low-energy impacts from such events as tool drops and kickups of roadway debris on structural strength, stiffness, and dimensional stability.

### Approach

- Characterize and model the durability behavior of a progression of three representative carbon-fiber composites, each with the same thermoset urethane matrix but having a different reinforcement preform: (1) continuous fiber, ±45° crossply; (2) continuous fiber, quasi-isotropic; and (3) random chopped fiber.
- Replicate on-road conditions in laboratory tests of each composite to generate durability data and models.
- Develop and publish durability-based design criteria for each composite.
- Shift focus to suitable thermoplastic composites, for which durability issues are generally more significant.

### Accomplishments

- Published report providing aspects of mechanical behavior of stitched T300 mat/urethane 420 IMR composite (ORNL/TM-2002-86).
- Completed planned durability tests of 3-mm-thick and 1.5-mm-thick chopped-carbon-fiber thermoset composite plaques.
- Published report on durability-based design criteria for a chopped-carbon-fiber automotive composite (ORNL/TM-2003-86).
- Published two reports on effects of temperature and environment on chopped-fiber automotive structural composites (ORNL/TM-2003/114 and ORNL/TM-2003/117).
- Published three technical papers.
- Completed initial baseline testing for the quasi-isotropic carbon-fiber poly (phenylene sulfide) (PPS) material.
- Fed data on chopped-carbon-fiber composite directly into the planning and analysis for the Focal Project III carbon-fiber body-in-white.

### Future Direction

- Investigate relationship between processing of PPS materials and crystallinity as well as relationship between crystallinity and material properties.
- Complete baseline characterization of carbon-fiber-reinforced PPS material.
- Complete durability assessment of carbon-fiber-reinforced PPS material.
- Publish durability-driven design criteria documents for representative thermoplastic composites suitable for automotive structural applications.

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## Introduction

Before composite structures will be widely used in automotive applications, their long-term durability must be assured. The Durability of Carbon-Fiber Composites Project at the Oak Ridge National Laboratory was established to develop the means for providing that assurance. Specifically, the project is developing and documenting experimentally based, durability-driven design criteria and damage-tolerance assessment procedures for representative carbon-fiber composite systems to assure the long-term (15-year) integrity of composite automotive structures. Durability issues being considered include the potentially degrading effects of cyclic and sustained loads, exposures to automotive fluids, temperature extremes, and incidental impacts from such things as tool drops and kick-ups of roadway debris. Research to determine the effect that these environmental stressors and loadings have on structural strength, stiffness, and dimensional stability is being conducted. The project is carried out in close coordination with the Automotive Composites Consortium (ACC).

It is envisioned that about 15% of the Focal Project III carbon-fiber-composite body-in-white will utilize directed continuous-fiber reinforcement architectures, while the remainder will employ random chopped-fiber reinforcement. The approach to investigating durability has thus been to address a progression of thermoset composites, each of which has the same urethane matrix:

- reference [±45]3S crossply composite,
- [0/90/±45]S quasi-isotropic composite, and
- randomly oriented chopped-carbon-fiber composite.

Characterization of the first two, continuous-fiber composites has been completed, and design criteria documents were published. In mid-FY 2002 the focus turned to chopped-carbon-fiber composites. Characterization of the randomly oriented chopped-carbon fiber composite was completed in FY 2003. Also in FY 2003 investigation of carbon-fiber-reinforced thermoplastic materials for structural automotive applications was initiated.

## Chopped-Carbon-Fiber Material

The ultimate goal for the Focal Project III carbon-fiber composite body-in-white is to achieve a fiber-volume fraction of 40% and a minimum thickness of 1.5 mm over 70% of the structure.

The ACC successfully molded chopped-carbon-fiber plaques meeting these Focal Project III requirements. The carbon fiber, chopped by the P4 machine in 50-mm lengths, was Zoletek Panex 33, seven-split 46K. The urethane matrix was Bayer 420 IMR, the same as in previous composites addressed by the Durability Project. Unfortunately, while the average properties of the 1.5–mm-thick plaques satisfied Focal Project III design requirements, the variability was excessive. It was originally planned to use 1.5-mm-thick plaques for durability studies. However, because of the larger than expected variability in the 1.5-mm material, the ACC instead fabricated and supplied 3-mm-thick plaques in March of 2002. These 3-mm-thick plaques, which exhibit reduced variability, have been adopted as the reference chopped-fiber material. The general project approach has been to first replicate on-road conditions in laboratory specimens to generate data to form the basis for developing correlations and models. These correlations and models were then used to formulate design criteria. Tests included the following:

- basic short-time tension, compression, and shear;
- uniaxial and biaxial flexure;
- cyclic fatigue, including mean stress effects;
- tensile and compressive creep and creep rupture;
- hole and crack effects;
- low-energy impact; and
- tension- and compression-after-impact.

In most cases, characterization of effects of temperature and fluid exposure were included in the test effort that involved almost 2,200 individual tests. Provided below are examples of results that were obtained under the test program. Although all tests noted above are not discussed below, additional information to that provided as well as results for tests not discussed can be obtained from report 2 in the “Publications“ section of this report.

Table 1 presents average in-air room-temperature (RT) baseline properties for the chopped-carbon-fiber composite.

**Table 1.** Tensile, compressive and shear properties

| Property | Tension | Compression | Shear |
|---|---|---|---|
| Modulus, GPa | 27.7 | 28.9 | 9.37 |
| Poisson’s ratio | 0.34 | — | — |
| Strength, MPa | 203 | 173 | 153 |
| Failure strain, % | 0.90 | 0.77 | 2.34[a] |

[a]Engineering shear strain.

From a durability standpoint, it is assumed that an automobile with a composite structure must last for 15 years and 150,000 miles. It is further assumed that during the 15 years the vehicle will actually be operated between 3,000 and 5,000 h. The design temperature range is taken to vary from a minimum of –40°C to a maximum of

120°C, with the possibility of the highest temperature occurring only during operation. The effect of thermal cycling is also a concern. The structures must support and resist a variety of dead and live loads. Also, the structures will be subject to common vehicle fluids and operating atmospheres, and design limits must take the resulting property degradation into account.

Temperature multiplication factors for relating at-temperature stiffness and strength to reference baseline RT values are provided in Figures 1 and 2, respectively. The basic time-dependent allowable stress, $S_o$, defined as two-thirds the minimum ultimate tensile strength (UTS) at RT, is about 52% the average UTS. Values of $S_o$ at other temperatures are obtained by multiplying the above RT $S_o$ value by the UTS factors provided in Figure 2. Prior thermal cycling (25 cycles from RT to 120°C to –40°C to RT) had a small effect on tensile strength and stiffness (≤3%), whereas compressive and shear stiffness dropped 5% and 11%, respectively.

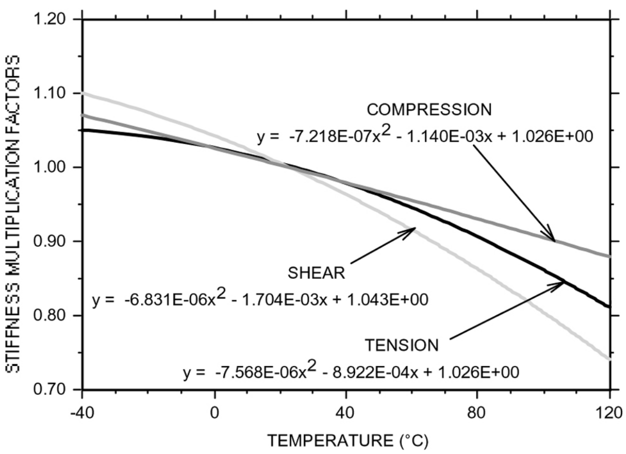


**Figure 1.** Temperature multiplication factors: stiffness.

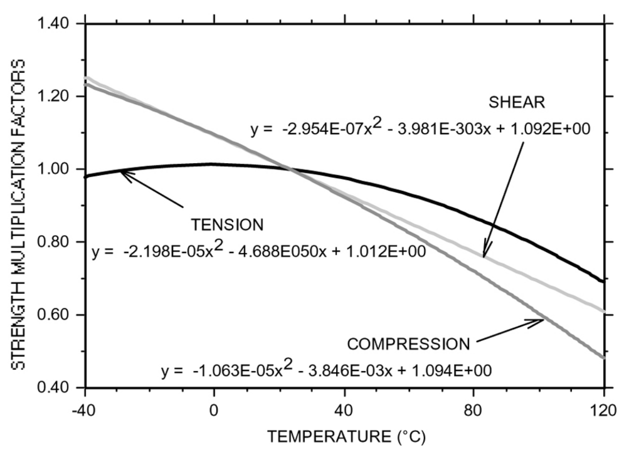


**Figure 2.** Temperature multiplication factors: strength.

In the durability assessment, it is assumed that structures will be subjected to common vehicle fluids and operating atmospheres, therefore design limits must take the resulting property degradation into account. Figure 3 presents effects of exposure to eight automobile fluids on apparent shear strength. Based on these results, plus results from weight gain and tensile properties vs exposure time, the fluids most extensively examined were distilled water and windshield washer fluid for exposure times of 1000 h and 100 h, respectively. To bound fluid effects on stiffness, a 4% reduction value is recommended.

Biaxial stress states exist in real structures, and bending is unavoidable at some locations. Both uniaxial and biaxial tests were performed at various temperatures and in both air and representative fluids, Figures 4 and 5, respectively.

In the simple beam tests, the modulus of rupture (maximum elastically calculated stress determined from simple bending theory) varied from 2.1 times the UTS at –40°C down to 1.3 at 120°C. Biaxial flexural

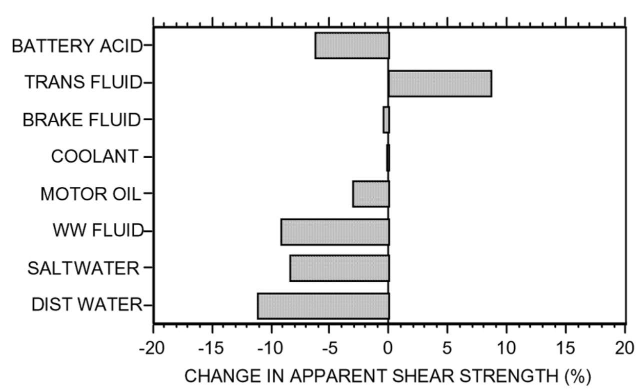


**Figure 3.** Effects of exposure on apparent shear strength.

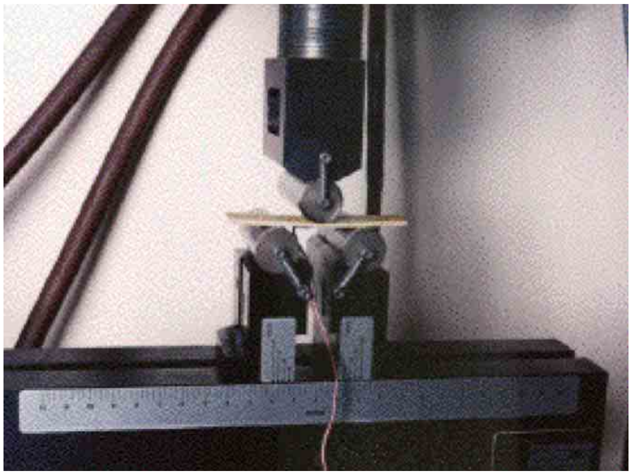

**Figure 4.** Uniaxial flexural test setup.

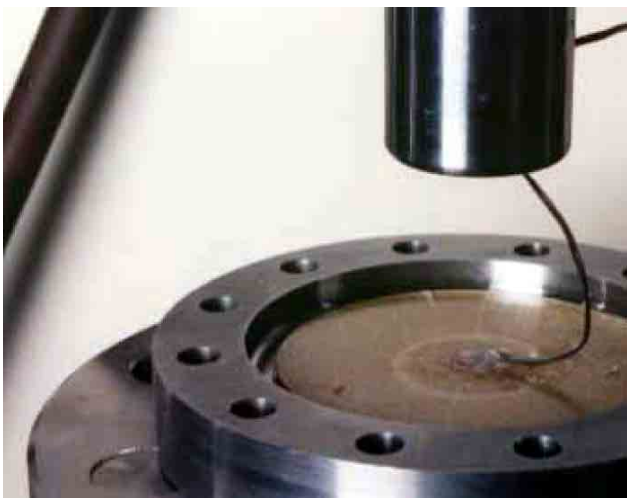

**Figure 5.** Biaxial flexural test setup.

strength results in which the average failure loads under different environments were compared to as-received RT results varied from 1.03 for 100-h presoak in windshield washer fluid down to 0.60 for 120°C exposure. Resulting failure data have been used in formulating a biaxial strength theory for the final design criteria. Table 2 provides a summary of strength multiplication factors relative to in-air RT values for short-time static loadings.

Fatigue S-N curves (stress vs number of cycles) for a number of loading conditions (e.g., tensile cycling, compressive cycling, and completely reversed stress cycling) were developed over the –40°C to 120°C temperature range and for specimens soaked for

**Table 2.** Summary of strength multiplication factors for short-time static conditions

| Stress state | 1000 h in distilled water | 100 h in windshield washer fluid | 120°C |
|---|---|---|---|
| Tension | 0.94 | 0.98 | 0.69 |
| Compression | 0.86 | 0.95 | 0.48 |
| Shear | 0.95 | 0.99 | 0.61 |
| Uniaxial flexure | 0.92 | 1.06 | 0.54 |
| Biaxial flexure | 0.90 | 1.03 | 0.60 |

100 h in windshield washer fluid or 1000 h in distilled water prior to testing. Figure 6 presents the single RT, ambient air design curve that has been developed. A special stress parameter, S, that combines the maximum stress, $S_{max}$, or in the case of a compressive cycle the absolute value of the minimum stress, $|S_{min}|$, with the alternating component of the stress, $S_a$, is used for all fatigue evaluations. The curve in Figure 6 is applicable to all cycles with mean stress of zero or greater. Fatigue strength multiplication factors have been developed to address situations where cycling into compression and a compressive mean stress is developed and to address temperatures other than RT and fluid exposures.

In the case of long-term sustained loadings—either those associated with the 3000-h or 5000-h operating life of a vehicle—time-dependent creep deformations may become an important consideration and

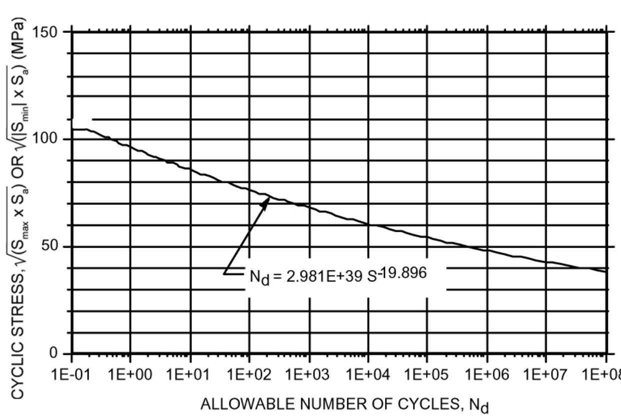


**Figure 6.** RT design fatigue curve.

need to be accounted for in the design analysis. Constant-load tensile creep deformation tests were performed in RT air, at elevated temperatures (70°C and 120°C), and in fluids (distilled water with a nominal presoak of 1000 h and windshield-washer fluid with a 100-h presoak). A limited number of compressive creep tests were also performed in RT air and elevated temperature. The approach was to develop an RT, in-air, tensile creep equation and to then develop creep multiplication factors to approximately characterize the effects of temperature, fluids, and compressive loadings in terms of RT, in-air, tensile response. Figure 7 presents tensile creep-rupture data and the design curve at 120°C compared to the RT curve. The design curve is 77% of the average curve as has been used with previous composites.

Damage tolerance is the ability of a material or structure to continue to perform its function in the presence of damage, which might occur in the form of initial flaws or as service-induced damage, such as that due to incidental low-energy impacts. Impact tests using a pendulum to represent tool drops and an air gun projectile to represent roadway kickups have been performed on plates of the chopped-carbon fiber material. Also, brick-drop tests were performed to determine the ability of the baseline tests to cover that event, which is of interest in the design of pickup truck boxes. In addition to baseline tests performed in ambient air, tests were performed at –40°C. Figure 8 presents the design curve that was developed for conservatively estimating damage from impactor mass and velocity.

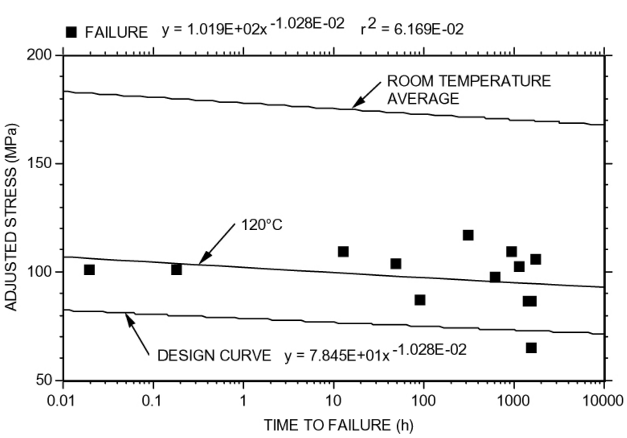


**Figure 7.** Tensile creep-rupture data and design curve at 120°C.

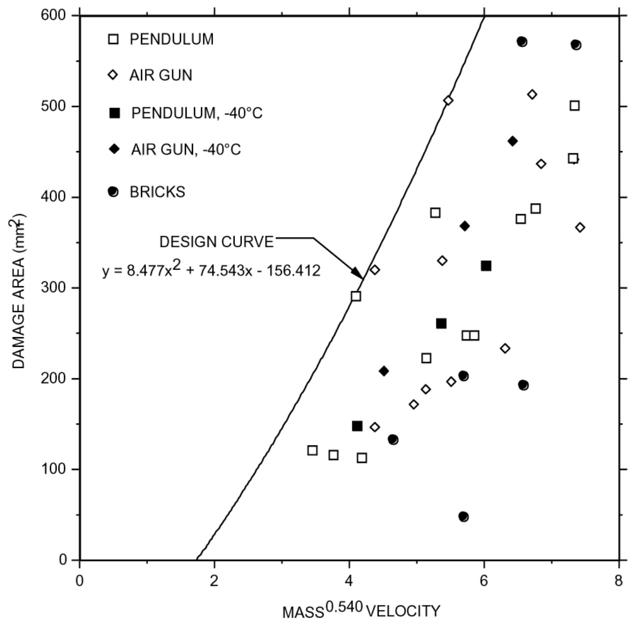


**Figure 8.** Design curve for estimating damage.

## Carbon-Fiber-Reinforced Thermoplastic Material Investigation

A quasi-isotropic carbon-fiber-reinforced PPS material has been selected as the next material to be investigated. The ACC has supplied 43 plaques, 510-mm by 610-mm by about 3-mm thick, for use in durability studies. Plaque reinforcement is symmetrical and consists of 16 plies of carbon-fiber unidirectional tape, [0/90/±45]2S. This is the first thermoplastic composite matrix material to be investigated under the durability program.

Processing conditions are very important because they affect the crystallinity of a semicrystalline polymer such as PPS. Crystallinity changes of thermoplastic materials can result in significant changes in the mechanical behavior of composites containing them, particularly with respect to matrix-dominated properties such as compressive strength and creep. For this reason the ability to precisely characterize the polymer crystallinity in a thermoplastic material becomes an important requirement. Unfortunately the precise processing procedure followed for preparing the PPS plaques is not available because the process is considered proprietary. Efforts are under way to obtain background information and data on the effects of processing on PPS crystallinity and the relationship between crystallinity and properties. Also, techniques are being identified for determining the crystallinity of the material supplied and determining the effect that specific durability issues addressed may have on the material crystallinity (e.g., 120°C thermal exposure, depending on the processing temperature, may increase material crystallinity).

Tests to evaluate specimen orientation effects and to establish baseline RT tensile and compressive properties of the PPS composite material have initiated. Figures 9 and 10 present the effect of specimen orientation on tensile strength and tensile modulus of elasticity (0° orientation is in long direction of plaque), respectively.

Additional baseline tensile and compressive properties have been obtained from specimens obtained from three of the plaques. Six specimens each in the 0° and 90° orientations were tested from each

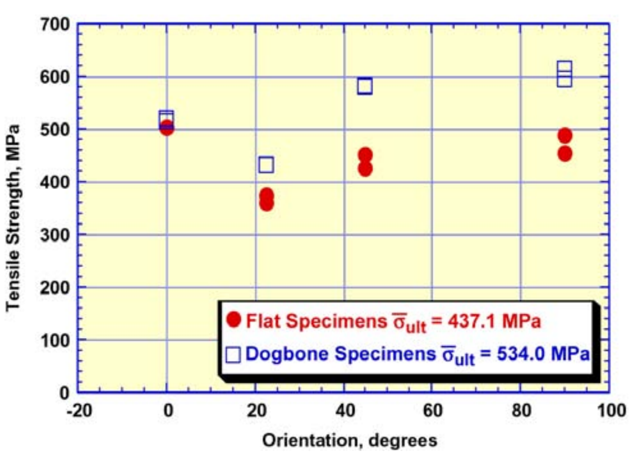


**Figure 9.** Specimen orientation and tensile strength.

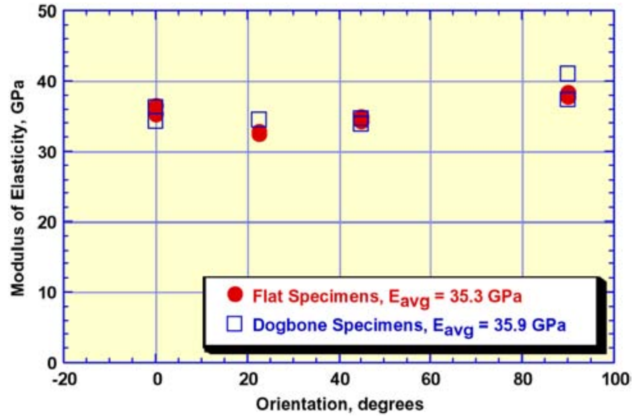


**Figure 10.** Specimen orientation and tensile modulus.

plaque. Table 3 presents preliminary average tensile and compressive properties obtained (tensile results also include those investigating specimen orientation effects).

Once the baseline shear properties are determined, testing to develop durability-based design criteria for the quasi-isotropic carbon-fiber reinforced PPS material will be initiated.

**Table 3.** Baseline tensile and compressive properties

| | *Tensile* | |
|---|---|---|
| Orientation | Strength (MPa) | Modulus (GPa) |
| 0° | 523 (16.6) | 36.9 (1.0) |
| 90° | 570 (24.8) | 40.1 (0.9) |
| | *Compressive* | |
| Orientation | Strength (MPa) | Modulus (GPa) |
| 0° | 314 (19.6) | 35.3 (1.3) |
| 90° | 353 (22.8) | 37.4 (1.1) |

## Summary

Recommended durability-based design properties and criteria have been developed for a quasi-isotropic carbon-fiber composite for possible automotive structural applications. The durability issues addressed included the effects on deformation, strength, and stiffness of cyclic and sustained loads, operating temperature, automotive fluid environments, and low-energy impacts. Guidance has been developed for design analysis, time-dependent allowable stresses, rules for cyclic loadings, and damage tolerance design guidance.

A quasi-isotropic carbon-fiber reinforced PPS material has been selected as the next material to be investigated. This is the first thermoplastic material to be investigated under the durability program. The ACC has supplied 43 plaques, 510-mm by 610-mm by about 3-mm thick, for use in durability studies. Plaque reinforcement is symmetrical and consists of 16 plies of carbon-fiber unidirectional tape, [0/90/±45]2S. Processing conditions for these plaques are very important because they affect the crystallinity of a semicrystalline polymer such as PPS, which can result in significant changes in the mechanical behavior of composites containing them. Because precise processing information on the plaques is not available due to the proprietary nature of the processing, the crystallinity of the material is being investigated. Initial tests to evaluate specimen orientation effects and to establish baseline RT tensile and compressive properties of the PPS composite material have been completed.

## Publications

1. S. Deng, X. Li, and Y. J. Weitsman, *Aspects of the Mechanical Behavior of Stitched T300 Mat/Urethane 420 IMR Composite*, ORNL/TM-2002/86, Oak Ridge National Laboratory, Oak Ridge, Tenn., October 2002.

2. J. M. Corum, R. L. Battiste, A. Ionita, M. B. Ruggles-Wrenn, and Y. J. Weitsman, *Durability-Based Design Criteria for a Chopped-Carbon-Fiber Automotive Composite*, ORNL/TM-2003/86, Oak Ridge National Laboratory, Oak Ridge, Tenn., May 2003.

3. J. M. Corum, R. L. Battiste, and M. B. Ruggles-Wrenn, "Low-Energy Impact Effects on Candidate Automotive Structural Composites," *Composites Science and Technology* **63**(6), 755–769, 2003.

4. S. Deng, X. Li, and Y. J. Weitsman, "Time-Dependent Deformation of Stitched T300/Mat/Urethane 420 IMR Cross-ply Composite Laminates," *Mechanics of Time-Dependent Materials* **7**(1), 41–69, 2003.

5. M. B. Ruggles-Wrenn, J. M. Corum, and R. L. Battiste, "Short-Term Static and Cyclic Behavior of Two Automotive Carbon-Fiber Composites," *Composites* Part A **34**, 731–741, Elsevier Ltd., 2003.

6. M. B. Ruggles-Wrenn, *Effects of Temperature and Environment on Mechanical Properties of Two Chopped-Fiber Automotive Structural Composites*, ORNL/TM-2003/114, Oak Ridge National Laboratory, Oak Ridge, Tenn., July 2003.

7. M. B. Ruggles-Wrenn, *Effects of Temperature and Environment on Mechanical Properties of Two Continuous Carbon-Fiber Automotive Structural Composites*, ORNL/TM-2003/117, Oak Ridge National Laboratory, Oak Ridge, Tenn., July 2003.